Particle mixing in gas-solid fluidised beds

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Chapter 3. Visual observations of individual particle behaviour in gas and liquid fluidised beds

3.1 Introduction

It is important for the modelling of processes occurring in fluidised beds to have knowledge about the nature of fluidised particles. How they move, how, if at all, they make contact and how segregation of atypical particle takes place.

Some indirect knowledge about these topics has been gained in the past. By introducing interparticle forces in the fluidised bed, e.g. by permanent magnetism of the particles (Agbim *et al.*, 1971), using a high gas humidity (Harnby *et al.*, 1987, Arai and Sugiyama, 1974) or using a non-volatile liquid to coat the particles (Seville and Clift, 1984), it was possible to change the fluidisation behaviour of the particles. Bubbling was suppressed and minimum fluidisation velocity increased. Therefore, it has been commonly accepted that the differences in fluidisation behaviour of Geldart's powder groups are not only due to hydrodynamic effects but are also influenced by interparticle forces. Moreover, measurements of the heat transfer in fixed and fluidised beds indicate that fluidised particles are in contact (Gamson, 1951, Mickley and Fairbanks, 1955). In spite of the above work giving clues about the behaviour of fluidised particles it has not been possible to conclusively determine the behaviour of individual particles in fluidised beds.

To investigate the particle motion directly, visual observations of the individual particles have been made at the riser wall of a cold model circulating fluidised bed (Rhodes *et al.*, 1992, Rhodes *et al.*, 1990). The interaction of group B with group C powders in the dense phase of a fluidised bed has also been examined in this way (Xia and Kwauk, 1987). By inserting fibre probes into fluidised beds it is possible to locally measure the bubble properties and particle velocities (Grace and Baeyens, 1986). Optic fibre probes have been used to determine particle velocities and concentrations in circulating fluidised beds (Zhou *et al.*, 1995, Zhou *et al.*, 1994, Wang *et al.*, 1993, Wang *et al.*, 1992, Horio *et al.*, 1992). Recently, it has become possible to visualise the individual particles within gas fluidised beds by means of special optical probes although the application has so far been limited to 'fast' fluidised beds where a rapidly moving dispersed and cluster phase have been viewed (Hatano and Kido, 1994, Li *et al.*, 1991, Takeuchi and Hirama, 1990).

In this study the behaviour of the individual particles in bubbling gas and in liquid fluidised beds is observed with an optical probe. The individual particle behaviour in these beds and the behaviour of jetsam particles in gas fluidised beds of group B binary mixtures of different density is reported. In a binary different density fluidised bed, visualisations were made by injecting bubbles and simultaneously studying the microscopic behaviour using the probe and the macroscopic behaviour using an X-ray technique. This gave additional information on possible disturbances created by the probe and on the reactions of the individual particles to rising bubbles. Although the results of the visualisations are mainly qualitative they are of great value in furthering the understanding of the nature of fluidised beds and the process of segregation.

3.2 Experimental

The optical probe consists of a series of lenses within a hollow tube surrounded by optical fibres connected to an adjustable halogen light source (Figure 3.1).

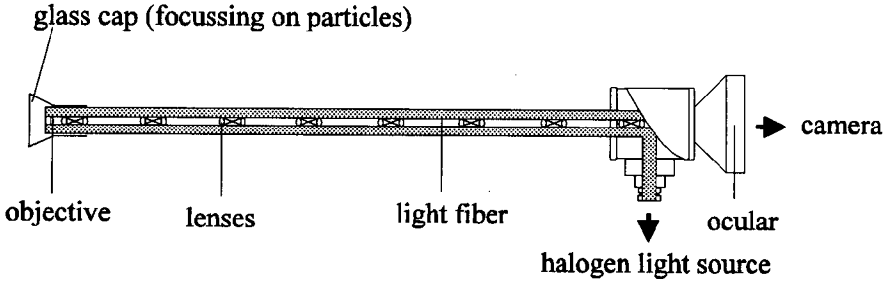

Figure 3.1 Schematic diagram of the probe.

An experimental type probe (7 mm diameter, 5 cm long) and a commercially available type (4 mm diameter, 17 cm long) both from Richard Wolf GmbH in Germany were used. A conical glass cap was used to prevent the particles from reaching the lens thus making it possible to focus on the particles. These devices had maximum diameters of 15 mm and 9 mm respectively. The images of the particles were collected by a CCD micro-camera (50 fields/s, 25 frames/s) connected to the ocular of the probe and using lenses with focal lengths of 27 mm and 75 mm respectively. An advanced video system and a monitor were used.

The probe was positioned horizontally in a fluidised bed column (6.6 cm I.D.) made of glass and fitted with a porous glass distributor. To ensure constant fluidisation conditions the relative humidity of the fluidising gas was controlled by means of a humidification apparatus (see Chapter 6). The feed for this apparatus was dry air with 10 % relative humidity coming from a compressor. The flow rate of dry air was set by means of an automatic controller and part of it was wetted in a water trickle-flow column filled with raschig rings. The dry and humid air streams were then mixed in a static mixer section, whereafter the relative humidity was measured and controlled automatically in a feed-back loop. Tap water was used for liquid fluidisation. Observations were made in bubbling gas-solid fluidised beds of 260-310 μm glass ballotini and of 210-260 μm bronze particles at velocities up to 0.25 m/s. Liquid fluidisation of the 260-310 μm glass ballotini was performed at superficial velocities between $0.99 \cdot 10^{-3}$ m/s and $5.85 \cdot 10^{-3}$ m/s (minimum fluidisation velocity $U_{mf} = 0.90 \cdot 10^{-3}$ m/s calculated with the Ergun (1952) equation). Other particle properties are given in Table 3.1.

The experiments using X-ray imaging were performed at the Department of Chemical Engineering at University College London. A description of the X-ray apparatus has been given by Rowe (1971). Bubbles were injected in a 105 - 115 μm copper, 125 - 350 μm alumina system at different 'background' superficial gas velocities ranging from U_{mf} to about 2 U_{mf}. The particle properties are given in Table 3.1. The solenoid valve was opened with frequencies between 1 Hz and 10 Hz and the pulse time of it was varied between 0.1 and 2.0 on its scale. Experiments were carried out in a rectangular alumina column of 15.2 cm x 19.55

cm and of 40.0 cm height using the endoscope and the X-ray apparatus of University College London simultaneously at various positions within the bed.

Table 3.1 Properties of the particles used in the experiments.

	d_{sv} [μm]	ρ_p [kg/m^3]	U_{mf} [m/s]
Endoscope observations			
260-310 μm glass ballotini	281	2500	0.067^1
	281	2500	$0.99\ 10^{-3}$
210-260 μm bronze particles	235	8750	0.19
X-ray observations			
105-115 μm copper particles	N.D.	8920	0.037
125-350 μm alumina particles	N.D.	1450	0.039

1: gas-solid fluidisation, 2: liquid fluidisation.
N.D. not determined

3.3 Results and observations

3.3.1 Freely bubbling group B powder

The probe was inserted in the bed both vertically and horizontally. With vertical insertion, the region below the probe was clearly seen to be defluidised. Horizontal insertion of the probe into the bed appeared not to cause disturbances in the bed by the probe tip (although bubble formation was observed near the bed wall where the probe entered). Particles were seen to move away from and towards the probe. Also segregation of heavier particles was observed. This indicates that the particles in front of the glass cap are normally fluidised and their motion not hindered by the probe. The observations obtained with the X-ray apparatus (see also section 3.3.3) showed negligible influence of the probe on the rising bubbles within the bed only in a few cases bubble splitting was observed when the apex of large bubbles touched the probe. It is therefore believed that the results obtained are representative for the particle behaviour in the bed.

Observations were made near the top of the bed (11.0 cm above the gas distributor) at different horizontal positions by simply pushing the probe further into the bed. The transition from the packed to the fluidised state when increasing the gas velocity was recognised by the passage of bubbles. A little rearrangement but no 'floating' of particles was observed before this transition. In the bubbling fluidised state the individual particles were largely immobile being in lasting contact with each other and they were only moving when disturbed by rising fluidisation bubbles. In Figure 3.2 a sequence of pictures shows the passage of a bubble near the top of the fluidised bed. The bright circle covering part of the screen is the result of light reflecting in the glass cap. The bright circle within each glass particle is also due to a reflection effect. As the bubble approaches, the particles are pushed up- and sidewards while the individual particles maintain their relative positions. In Figure 3.2A the moving particles appear as unsharp streaks due to the relatively long exposure time of the camera. In Figure 3.2B the empty internals of the bubble appears dark. Figure 3.2C shows the particles just after the bubble passage where they are no longer moving upwards. At this point the voidage is still higher than in the bulk phase as a whole. Figure 3.2D shows the particles settling to the normal bulk voidage. They then stay essentially immobile until a new bubble approaches. The observed behaviour appeared consistent with particle movement as in a plastically deforming structure without much mobility of the individual particles. The momentum transfer in the bed appeared to be via particle-particle contacts which were lasting and not collisional. This is, unfortunately, less evident from the still pictures than from the moving film.

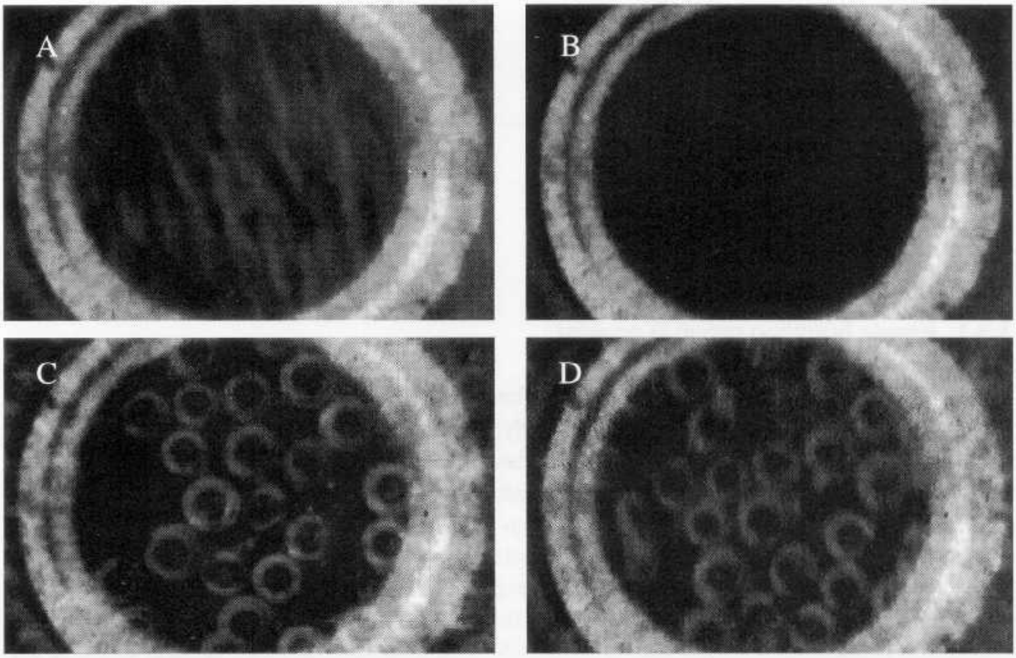

Figure 3.2 *Sequence of pictures showing the passage of a bubble (Commercial probe: elapsed time between two pictures is 3/25 s, H_{bed} = 17 cm, H_{probe} = 11.0 cm, U_G = 0.08 m/s):*
***A**: moving particles appear as unsharp streaks,*
***B**: dark view of the empty internals of a bubble,*
***C**: particles moving slowly upwards after a bubble,*
***D**: particles returning to the dense phase voidage.*

Near the wall, where no bubbles rose, the particles were moving downwards while largely maintaining their position within the arrangement of touching particles. Some deficiencies (holes of dimensions of the same order as the particles) were always observed both near the top as near bottom of the bed. Such a deficiency has been indicated in Figure 3.3B.

In order to observe the particle behaviour close to a 'distributor plate' bronze powder was added to the bubbling fluidised bed of glass ballotini at gas velocities below its own minimum fluidisation velocity. The bronze segregated to the bottom of the bed and acted as a gas distributor. Compared to higher up in the bed where the particle only move in response to the rising bubbles the particles moved continuously but on a smaller scale near the bronze powder distributor. As the voidage was clearly higher the individual particles could move and rearrange their relative positions to a much greater extent. Although the mobility is higher the particles still seem to be in lasting contact with each other.

3.3.2 Observations of segregating bronze particles

In a bubbling binary mixture of glass and bronze particles the individual bronze particles appeared not to be supported in the gas stream but to be lying on a supporting structure of touching glass particles. The bronze particles segregated by falling through defects when provided with the opportunity as a consequence of a disturbance created by a rising gas bubble. A sequence of frames showing the segregation of a bronze particle close to a defluidised layer is shown in Figure 3.3.

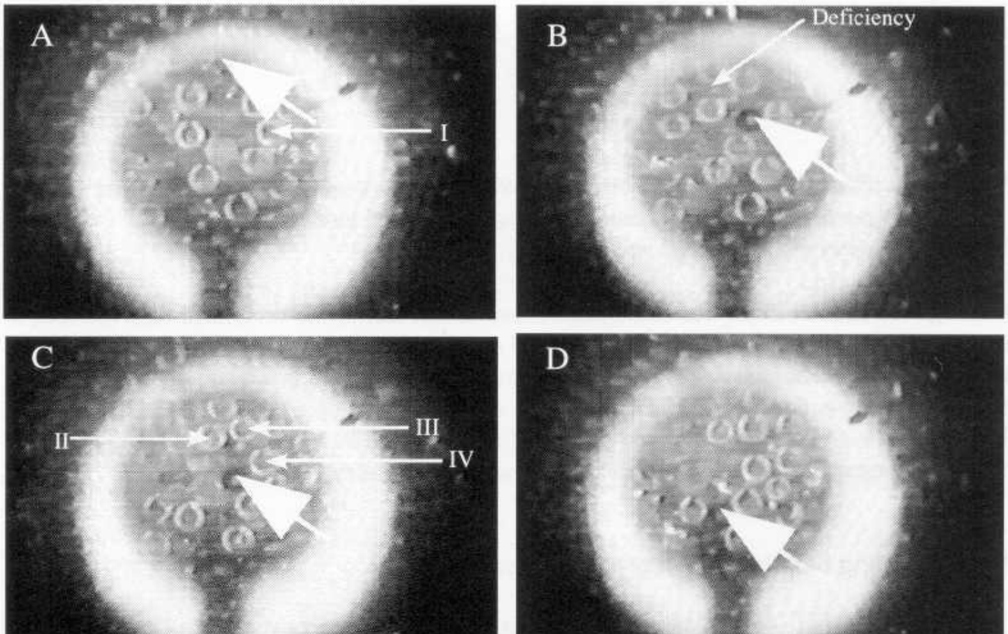

Figure 3.3 Sequence of pictures showing the segregation of a bronze particle near the distributor (Experimental probe: elapsed time between two pictures is 3/25 s, $H_{bed} = 20\ cm$, $U_G = 0.08\ m/s$*)*

The movements of the indicated bronze particle and that of several glass particles (I - IV) were tracked. The results are shown in Figure 3.4. It can be seen in this figure that the motion of the jetsam particle is downwards, while the four bulk particles move arbitrarily around in the in field of view.

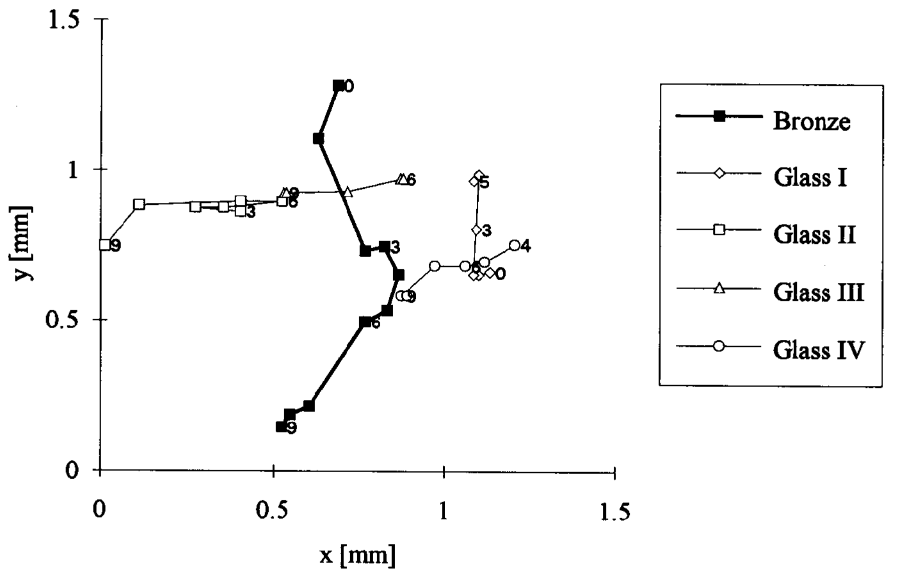

Figure 3.4 Particle tracking showing the movement of a bronze and of various glass particles which are indicated in Figure 3.3. The numbers in the figure correspond to the frame numbers (25 frames/s).

Although the bubbles in the bed could not be seen clearly due to the very small field of view, it appeared that a jetsam rich region exists behind a bubble. This agrees with observations of the mixing in two-dimensional fluidised beds (Rowe *et al.*, 1972) and it is now commonly accepted that jetsam rises in the wake of the bubbles. By adding a large amount of bronze at the top of the bed, it is even possible to defluidise part of the bed as a cluster of bronze particles forms which only segregates by increasing the gas velocity considerably.

3.3.3 Injecting bubbles in a segregated layer

Simultaneous visualisations with the endoscope and the X-ray apparatus (Figure 3.5) showed the particle movements in response to a rising bubble injected in a segregated layer of copper. On top of this segregated layer lay the alumina particles, the background superficial gas velocity was 3.9 cm/s which is the U_{mf} of the alumina. The endoscope tip is positioned in the middle of the bed at a height of 12 cm above the gas distributor. In figure 3.5 it is seen that at t = 1/25 s the bubble pushes copper out of the layer and that the gas void is covered with

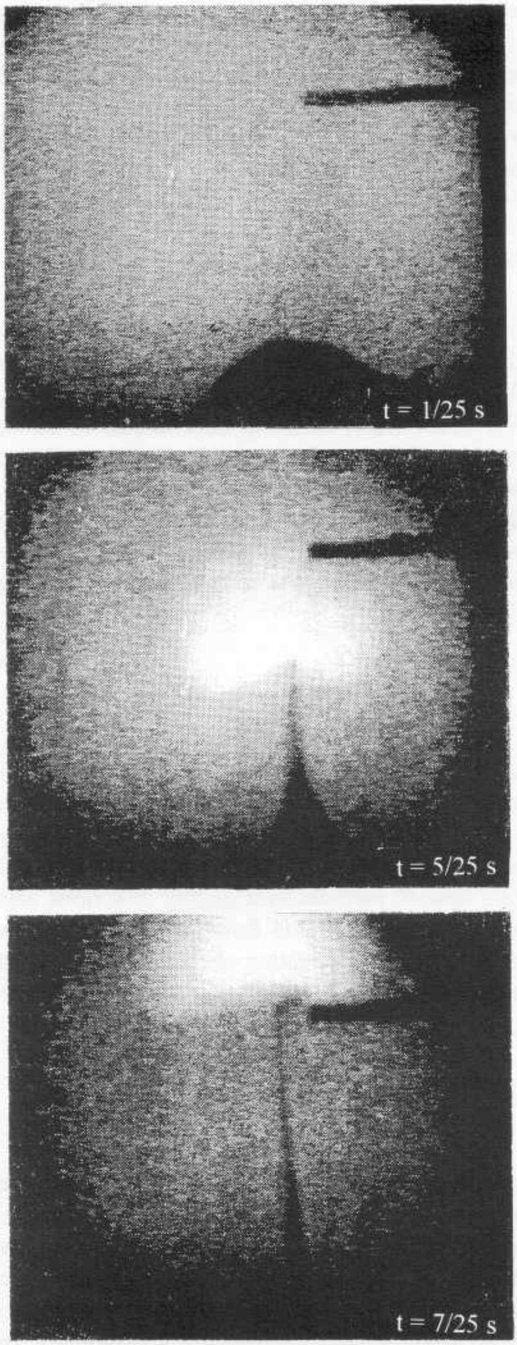

Figure 3.5 *Visualisations with the X-ray apparatus.*

copper particles. At this time alumina particles at the probe tip are not yet moving. A small part of the copper was present within the bubble while part of the copper is under the bubble. Part of the copper is transported upwards as a drift behind the bubble. At t = 5/25 s the centre of the bubble was at a height of approximately 9 cm. From the corresponding image obtained with the endoscope it is seen that the alumina particles are moving in response to the approaching bubble. While rising, the bubble seemed to get larger in diameter as it flattens out. At the same time the trail of the copper particles behind the bubble got thinner and longer and attained a conical shape. Copper was clearly visible in the wake of the bubble but was not uniformly distributed. The conical shape of the trail behind the bubble got thinner but was still connected to the copper present in the wake of the bubble. Through the endoscope the passage of the bubble void was observed as dark. At t = 7/25 s the bubble was at 15 cm and copper was still visible in the wake. The next picture showed that the bottom of the bubble is still visible on the X-ray while the bubble nearly disappeared from the image. The trail behind the bubble stayed connected to the wake but stretched out and got thinner. The copper in the wake of the bubble is flattened out but did not seem to have decreased in total size. Endoscope observations still showed particle movements behind the bubble. In the period from 9/25s to 12/25 s it could be seen that the drift pattern remained at the same position but it got thinner and less distinctive and also the bottom of the trail got thicker. This indicates that transport of copper particles downwards takes place. The endoscope showed that at $t = 12/25$ s the particles are not moving anymore near the probe tip.

Additional bubble injection experiments were carried out at the higher background superficial gas velocity of 4.9 cm/s. Opening frequency of the solenoid valve was set to 10 Hz. The X-ray pictures showed bubbles already present within the bulk of the material generating disturbances and pushing aside the drift trails behind the injected bubbles.

3.3.4 The effect of the humidity on the fluidisation of group B powder

The effect of the humidity of the fluidising gas on the 260-310 μm glass ballotini was also investigated. From literature it is known that increasing the gas relative humidity increases the interparticle forces (Harnby *et al.*, 1987, D'amore *et al.*, 1979). When the relative humidity of the fluidisation gas was increased from 45% in the freely bubbling experiments to about 65%, the particle mobility decreased and the number of deficiencies seemed to increase. When the humidity was increased above 65% the bubbling ceased and the particles became immobile. Sometimes it was observed that a few particles were blown away from a particular position after which a stable channel was formed there. The powder thus defluidised at high relative humidities exhibiting a more cohesive behaviour which is sometimes referred to as exhibiting 'group C behaviour'.

3.3.5 Liquid fluidisation of glass ballotini

Observations made in liquid fluidised beds of glass ballotini showed a bubbleless expansion of the bed and an increase in voidage while increasing the liquid flow rate from $0.99 \cdot 10^{-3}$ m/s to $5.85 \cdot 10^{-3}$ m/s. Contrary to gas fluidised beds where particles are largely immobile in the absence of fluidisation bubbles, near the top of the liquid fluidised bed (11.0 cm above the distributor) intermittent streamlike flow of particles in changing directions was observed, particularly at higher fluidisation velocities. In the stream flow individual particles were observed to move relatively to other particles and to the direction of the stream. In spite of the higher mobility, the particles appeared to make lasting contacts which at the lower fluidisation velocities resulted in the formation of intermittent, largely immobile, arrangements.

3.4 Discussion

The observation of constantly touching particles in the fluidised state agrees with the assumptions of Gamson (1951) and Mickley and Fairbanks (1955) which they used to correlate heat and mass transfer measurements performed in fixed and fluidised beds. We find that this arrangement of touching particles supported in the gas stream is present also when cohesion forces between the particles are negligible compared to other forces. Frictional resistance to sliding also appears to stabilise the formation of an arrangement of particles of low mobility. In this kind of arrangement the mobility of the individual particles depends on the frequency with which they are provided with the opportunity to leave their position which again depends on the disturbances caused by the fluidisation bubbles, on the voidage and the frictional and (for cohesive powders) the cohesive forces between the particles.

In gas fluidised beds of group B powder with relatively insignificant interparticle cohesion forces, the observed limited mobility of the individual particles thus appears to be a consequence of the frictional resistance to sliding. Near the top of the bed the voidage is low and bubbles are large which results in a low individual particle mobility. Near the gas distributor the voidage is higher and the smaller bubbles generate frequent small-scale disturbances which causes a higher particle mobility. Increasing the interparticle forces by humidification of the fluidising gas creates cohesiveness of the powder which decreases the particle mobility and results in channel formation and a packing of completely immobile particles with dense and less dense regions. In liquid fluidised beds the particle mobility is quite high which is thought to be due to the lubrication effect of water decreasing the frictional resistance to sliding and also due to the more severe effects of flow perturbations in the fluidising fluid in this kind of bed. Such a lubrication effect of water was also found in bulk density measurements (Harnby *et al.*, 1987).

The X-ray observations of bubble injection experiments in a different density mixture showed wake and drift particle mixing similar to what has been observed by Rowe (1971) who used a binary mixture of particles differing less in density than this present ones. The bubble wakes in our system are not completely filled with copper while his were. As suggested by Hoffmann *et al.* (1993) the larger exchange between the wake and bulk phase jetsam in our system is probably due to larger density differences in the components. However, it should be emphasised that the mixture investigated represents the limited situation of a fully demixed bed with maximum density differences. While becoming mixed the density differences between wake and bulk decreases largely.

3.5 Conclusions

From the endoscope and X-ray observations in the group B gas fluidised beds and in liquid fluidised beds it can be concluded that the particles in the dense phase are in lasting contact most of the time, being largely immobile in a structure-like arrangement while being fluidised. This arrangement seems to be the result of interparticle friction forces and, in cohesive powders, it is probably stabilised by interparticle cohesion. Also, as a result of these forces the individual particle mobility is lower in gas than in liquid fluidised beds. In a gas fluidised group B powder the particle mobility decreases with increasing powder cohesiveness obtained by increasing the gas relative humidity. Particle movements appeared to be induced by rising bubbles. The particle mobility decreases towards the top of the bed, because the voidage is lower and disturbances by bubbles are less frequent and of larger scale. Momentum transfer in the particle phase was observed to be via lasting interparticle contacts.

In the gas fluidised bed containing a binary mixture, heavier particles appear to be lying on a structure of touching glass ballotini. When provided with the opportunity through disturbances caused by rising bubbles they move through deficiencies and settle towards the bottom of the bed.

Notation

d	diameter	m
H	height	cm
U	superficial velocity	m/s
t	time	s

Greek symbol

ρ	absolute density	kg/m^3

Sub- and superscripts

bed	bed
G	gas
mf	at minimum fluidisation
p	particle
probe	probe
sv	average surface volume

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